IN THE UNITED STATES PATENT AND TRADEMARK OFFICE



REQUEST FOR FILING (RULE 53(b)(1))

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			For Design or	Utility Applica	tions	~~~
Rule	53(b)(1) PATENT	<u> L'APPLICATION</u>	<u>:</u>			(DO NOT USE FOR CIEST
Li	Continuation) application u	nder 37 CFR 1.53(b)(1)		ם,
\boxtimes	Divisional)	11001 07 01 11 1:00(b)(1)		
applio	cation under 37 C of pending prid	CFR 1.53(b)(1) or application of		Group Art	Unit: <u>1771</u>	
	P	or approaudit of		Examiner:	_RAIMUND, C.	
Inven	itor(s): VAN	DER LOO			TO MINIONED, O.	
Parer	nt Appln. No.:	08	995,754	Atty. Dkt.	PM 273691	8274US/CON/WO/DIV
Parer	nt Filed: DECEN	Series Code û	Serial No. û		New M#	Client Ref
		MBER 22, 1997 SEPTEMBER 15	, 2000			
Title:	BALLISTIC-RI OF THE MOU	ESISTANT MOL ILDED ARTICLE	ILDED ARTICLE AND	A PROCESS	S FOR THE MAN	UFACTURE
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Wash	ington, DC 2023				September 21, 20 iter No. 244464	
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Sir:						
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i Aud	10 61	rect the above-re	equested filing today:			
1	Attached is a c	opy (which mus	st be filed) of the prior	r application,	including:	
	57					
13 123	☒ Abstract☒ Specificati	on and alaims (C	22 pages) (must be at	4		
		(must be attach	ed if originally filed)		(s)/set: 1 set	informal:
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111	Always X one b	ox, only:				
(1) (2)	⊠ Copy of <u>Si</u>	gned declaration	or oath as originally t	filed in prior a	pplication attache	<u>ed</u>
(کے)	☐ <u>NO</u> deciara	audii or iee is en	closed; therefore, this	is a filing und	der Rule 53(f).	
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2.	☐ This app	lication is hereby	filed by less than all	of the invento	rs named in the	orior application. Petition is
	nereby n	nade requesting	deletion as inventor(s	i) of the follow	ing who is/are n o	of the
	invention	i being claimed i	n this application (DÈ	LEIE IHEFC	DLLOWING INVE	NTOR(S)):
	1.			2.		
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	7.			8.		
2.5	THE INVENTOR	D(Q) EOD TUIC I	NEW APPLICATION I	O(4DE)	-	
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3.	The section 11					
J.	application and i	sure of the prior is hereby incorpo	application is conside orated therein by refer	ered as being ence thereto.	part of the disclo	sure of the accompanying

4.	oxtimes Priority is claimed under 35 U.S.C. 119/365 based on filing in <u>THE NETHERLANDS</u> of
	(country)
	(1) 1000598 20 JUNE 1995 (2)
	\sim (4)
	(5)(6)
	a. (No.) Certified copy/copies attached
	b. 🖂 Certified copy/copies previously filed on DECEMBER 22, 1997 in
	U.S. Application No. 08/995,754 , filed on DECEMBER 22, 1997.
	c. ☐ Certified copy/copies filed during International stage of PCT/ NL96/00247.
4.	(a) Unimediate Domestic priority is claimed from / filed
	(b) Benefit is claimed of Provisional Application No. 60/, filed
5.	Prior application is assigned to <u>DSM N.V.</u>
	by assignment recorded APRIL 15, 1998 Reel 9098 Frame 0912.
6.	(Date) Attached is the following number of Assignments (including original and all later suggestive area by
6.	Attached is the following number of Assignments (including original and all later successive ones by different assignors): _1 and respective new Cover Sheets. (Do NOT file old cover sheets.)
STATE OF THE PARTY	
131	(Assignments in parent must be refiled with new Cover Sheets in this continuing application if you
	want it/them recorded against the continuing application.)
	Please return the recorded Assignment to the undersigned.
7	The power of attorney in the prior application is to KENDREW H. COLTON, 20, 269
11	The power of attorney in the prior application is to KENDREW H. COLTON, 30,368
A Comment of the Comm	(Name and Reg. No.) whose current address is as in item 8 below.
121 121 133	
-š	a. Recognize as associate attorney
income in	(Name, Reg. No. and Address)
	-
8.	Address all future communications to Intellectual Property Group
	of Pillsbury Madison & Sutro LLP, Ninth Floor, East Tower 1100 New York Avenue, N.W., Washington, D.C. 20005-3918
9.	
9.	Amend the specification by inserting before the first line the sentence:This is a continuation division of Application No. 08/995,754, filed DECEMBER 22 1997
	Series code ît ît serial no
	ALLOWED ON JULY 28, 2000, WHICH IS A CONT. OF PCT/NL96/00247 FILED JUNE
	14, 1996.
9.	(a) Amend the specification by inserting before the first line:This application claims the benefit of
	Provisional Application No. 60/, filed
10.	It has been recently determined that this new continuing application is entitled to small entity status.
	nence.
	(No.) Verified Statement(s) establishing "small entity" status under Rules 9 & 27 were/are: filed in above prior application (and hence applicable hereto)
	attached.
11.	Petition to extend the life of the characteristic and the characteri
(<u>one</u> box	Petition to extend the life of the above prior application to at least the date hereof is being concurrently filed in that prior application (Use Form PAT-111).
(<u>must</u> be	⇒ U was previously filed in that prior application (Check length of prior extension)
(X'd)	is not necessary for copendency (Double check before X'ing this box).

12	INFORMATION DISCLOSURE STATEMENT: Attached is Form PTO-1449 listing all of the documents cited by Applicant and the PTO in the parent application(s) relied upon under 35 USC 120 and referenced in item 9 above. Per Rule 98(d) copies of those documents are not required now. Please consider those documents and advise that they have been considered in this new application as by returning a copy of the enclosed Form PTO-1449 with the Examiner's initials in the left column per MPEP 609.							
13.		Attached is a Ru	le 103(a) Petitio	on to Suspend A	Action.			
14.		except for correct	ition of imprope	r multiple depe	ndencies or car	culation: (Do no ncellation of whole §§ 506 and 607;	e claims or mu	ltinle
		Please cancel claim 11, line 1 of Claims 13 and 14 Claim 15, line 1, Claim 16, line 1, Claim 17, line 1, Claim 18, line 2,	delete "8 or". 4, line 1, chang change "any or change "any or change "any or	ne of the claims ne of the claims ne of the claims	8-14" toclain 8-15" toclain 8-16" to clair	า 9 ท 9) .	
To the No		>- <u>CLAIMS AS I</u> Dox 1A <u>2</u> is X'd, do	FILED AND CH	FILING LOWING FILIN ANGED BY PF	G FEE IS BASI	ED ON <u>MENDMENT IN I</u>	ITEM 14<-<-	<-
America	bu <u>PTO</u> : PL	t leave lines 15-22	2 and 27-32 <u>bla</u>		VE IS X'D.			
Value						Large/Small Entity		Fee Code
15.	Basic Fili	ng Fee		Desi	an Application	\$310/\$155		106/26
Дþ.	Basic Fili	ng Fee	· · · · · · · · · · · · · · · · · · ·	<u>Not</u> Desi	gn Application	\$690/\$345	+690	101/201
17.	Total Effe	ective Claims	11	minus 20 =	0	x \$18/\$9	+0	103/203
_		ent Claims	1	minus 3 =	0	x \$78/\$39	+0	102/202
19.	If any pro	<u>per</u> multiple depe	ndent claim (igr	nore improper)	is present,	\$260/\$130	+0	104/204
<u> 20.</u>	0. Subtotal = \$690							
21.	1. If "petition" box 13 above is X'd, add petition fee							
21A	A. If box 6 above is X'd, add Assignment recording fee \$ 40							
22.	TOTAL EU INO EEE ATTAQUED							
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	(carry forward to Item 31)							
23.	·							
24.	⊠ Pr	eliminary Amendr	nent <u>attached</u> (to be entered <u>a</u>	<u>fter</u> assigning A	ppln. No.)		
25.	☐ Th	e following PREL	IMINARY AME	NDMENT is to	be entered <u>afte</u>	gassigning Appln	. No.:	

ADDITIONAL FEE CALCULATION FOR PRELIMINARY AMENDMENT PER BOXES 24/25

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						Large/Smal	l Entity		File Code
27.	Total Effective Cl	aims <u>*9</u>	minus ** _2	20 =	<u>0</u> x	\$18/\$9	=	\$_0	(103/203)
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29.	If amendment en first time, add (pe	ters proper mul er application)	tiple dependent	claim(s)	into this ap	plication for	the '\$130	+_0	(104/204)
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33.	*If the entry in this space i	s less than a entry in th	ne next space, the "Pres	ent Extra" re	sult is "0"				
	**If the "Highest number pr	eviously paid for" (see	item 17 above) is less th	nan 20, write	20" in this space				
35.	If the "Highest number prev	viously paid for" (see ite	em 18 above) is less tha	n 3, write "3"	n this space				
	Our Deposit Acco	ount No. 03-39 ⁻ 30268		2004					
interesting in the control of the co	ou order NO.		Z/:	3691 M#	*				

CHARGE STATEMENT: Upon the filing of a Declaration pursuant to Rule 60(b) or 60(d), the Commissioner is hereby authorized to charge any fee specifically authorized hereafter, or any missing or insufficient fee(s) filed, or asserted to be filed, or which should have been filed herewith or concerning any paper filed hereafter, and which may be required under Rules 16-18 (missing or insufficient fee only) now or hereafter relative to this application and the resulting Official document under Rule 20, or credit any overpayment, to our Account/Order Nos. shown above for which purpose a duplicate copy of this sheet is attached.

This CHARGE STATEMENT <u>does not authorize</u> charge of the <u>issue fee</u> until/unless an issue fee transmittal form is filed.

Pillsbury Madison & Sutro LLP Intellectual Property Group

1100 New York Avenue, NW Ninth Floor Washington, DC 20005-3918 Tel: (202) 861-3000 KHC/amp

Sig:

By Atty: Kendrew H. Colton

Reg. No.

30368

_____ Fax:

(202) 822-0944

Tel:

(202) 861-3606

NOTE No. 1: File this Request in <u>duplicate</u> with 2 postcard receipts (PAT-103) & attachments NOTE No. 2: Is extension in parent necessary for copendency? <u>DOUBLE CHECK Item 11 above.</u> If yes, printout Pat-111 and head it in parent.

Atty./Sec.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re PATENT APPLICATION of

VAN DER LOO Group Art Unit: 1771

Divisional of Appln. No.: 08/995,754 Examiner: C. Raimund

Parent Filed: December 22, 1997

Title: BALLISTIC-RESISTANT MOULDED ARTICLE AND A PROCESS FOR THE MANUFACTURE

OF THE MOULDED ARTICLE

PRELIMINARY AMENDMENT

Hon. Commissioner of Patents and Trademarks Washington, DC 20231

Sir:

Please enter this Preliminary Amendment.

IN THE CLAIMS:

Please cancel claims 9, 10 and 11 without prejudice or disclaimer.

Please amend claims 12-18 as follows:

12. (Amended) Process according to claim 19, wherein [11, characterized in that the] monolayer packages, too, are compressed and cooled at a pressure of at least 5 MPa.

VAN DER LOO -- Application No. 08/995,754

- 13. (Amended) Process according to claim 19, wherein said [9, characterized in that the specified] pressure is at least 7 MPa.
- 14. (Amended) Process according to claim 19, wherein said [9, characterized in that the specified] pressure is at least 10 MPa.
- 15. (Amended) Process according to claim 19, wherein said [9, characterized in that the] reinforcing fibres in the monolayers have a cross-section aspect ratio of at most 3.
- 16. (Amended) Process according to claim 19, wherein said [9, characterized in that the] monolayer has been obtained by impregnating the reinforcing fibres with an aqueous dispersion containing the plastic matrix material.
- 17. (Amended) Process according to claim 19, wherein said [9, characterized in that the] monolayer has a fibre weight of between 50 and 150 g/m².
- 18. (Amended) Ballistic-resistant moulded article obtainable <u>from the semi-manufactured article according to claim 19</u>, [according to the process of any of the claims 8-17,] characterized in that the moulded article has a SEA of at least 75 Jm²/kg on impact of a 7.62 Mild Steel Core P.S. Ball M1943 bullet.

Please amend claim 19 as follows:

19. (Amended) A semi [Semi]-manufactured article [according to the first step in the process according to claim 9] for the manufacture of a ballistic-resistant moulded article, said semi-manufacture article being obtained by forming a stack of cross-layered monolayers, said stack having an areal density of from 0.25 to 5 kg/m², and compressing said stack at an elevated pressure and cooling said stack at a given first compressive pressure which is at least 5MPa, [according to any of the claims 9-16, characterized in that the areal density of the] said semi-manufactured article [is] having an areal density between 0.25 and 5 kg/m².

Please add the following new claim as follows:

--20. A semi-manufactured article according to claim 19, wherein said stack comprises consolidated monolayer packages, containing from 2 to 18 monolayers that are placed cross-wise so as to form cross-layered monolayers.--

REMARKS

The above claims are presented for examination. Amende claim 19 avoids improper multiple dependencies. New claim 20 is based on former claims. Amended claims 12-18 are now amended to depend from amended claim 19.

VAN DER LOO -- Application No. 08/995,754

This application is thought to be in condition for allowance and Notice to the same is respectfully requested.

The attached PTO 1449 Form lists references for the Examiner's independent review and consideration. It is thought that these references are available to the Examiner as they were of record in the parent application. The Examiner is respectfully requested to acknowledge the independent consideration of these references.

Respectfully submitted,

PILLSBURY MADISON & SUTRO, LLP

y: /xm (ll

Kendrew H. Colton Reg. No. 30368

Tel. No. (202) 861-3606 Fax No. (202) 822-0944

KHC:amp 1100 New York Avenue, NW Ninth Floor, East Tower Washington, DC 20005-3918

APPLICATION UNDER UNITED STATES PATENT LAWS

A. (D1 - D7	77.5.4		
Atty. Dkt. No.	PM 273691 (M#)	-	
Invention:	VAN DER LOO		
Inventor (s):	BALLISTIC-RESISTANT MO MANUFACTURING ARTICL	DULDED ARTICLE AND E	O A PROCESS FOR THE
			Pillsbury Madison & Sutro LLP Intellectual Property Group 1100 New York Avenue, NW Ninth Floor Washington, DC 20005-3918 Attorneys Telephone: (202) 861-3000
No Andrews			This is a:
			Provisional Application
			Regular Utility Application
			Continuing Application ☑ The contents of the parent are incorporated by reference
			PCT National Phase Application
			Design Application
			Reissue Application
			Plant Application
			Substitute Specification Sub. Spec Filed in App. No. /
			Marked up Specification re Sub. Spec. filed In App. No /

SPECIFICATION

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5 BALLISTIC-RESISTANT MOULDED ARTICLE AND A PROCESS FOR THE MANUFACTURE OF THE MOULDED ARTICLE

resistant moulded article containing a compressed stack of monolayers, with each monolayer containing unidirectionally oriented reinforcing fibres and at most 30 wt.% of a plastic matrix material and with the fibre direction in each monolayer being rotated with respect to the fibre direction in an adjacent monolayer. The invention also relates to a process for the manufacture of the moulded article. The ballistic-resistant moulded article may be used in, for instance, helmets, as inserts in bullet-proof vests, as armouring on military vehicles and in ballistic-resistant panels.

on military vehicles and in ballistic-resistant panels. A ballistic-resistant moulded article of this type is disclosed in WO 89/06190. The known ballisticresistant moulded article affords fairly good protection against impacts of projectiles such as shrapnel or bullets. The level of protection is quantified by means of the Specific Energy Absorption (SEA), a measure of the amount of energy that can be absorbed by a moulded article on impact of a projectile per unit areal density of the moulded article. Fairly good protection by the known moulded article on impact of a bullet is intended to mean an SEA value of from about 30 Jm²/kg to maximum 65 Jm²/kg. The March 1992 "Ballistics" leaflet of Messrs Allied-Signal Inc., Petersburg, Virginia, quotes as highest SEA level an SEA of 65 Jm²/kg on impact of a 7.62 x 39 Mild Steel Core P.S. Ball M1943. This SEA level has been achieved after years of optimization and was regarded by ballistics experts as being very high.

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Nevertheless, there continues to be a great need for ballistic-resistant moulded articles that can offer increased protection against impacts of projectiles of various kinds, especially against projectiles in the form of bullets.

The inventors of the present invention have found that this object is achieved by the density ρ_P of the compressed stack in the ballistic-resistant moulded article being at least 98.0% of the theoretical maximum density.

. The theoretical maximum density ρ_{th} is calculated using the following formula:

 $\rho_{th} = \rho_f m_f + \rho_k m_k$ where

 $\rho_f = \text{density of the fibres}$

 ρ_k = density of the plastic

 $m_{\text{f}} = \text{mass}$ fraction of the fibres in the compressed stack

 m_k = mass fraction of the plastic in the compressed stack

Surprisingly, a ballistic-resistant moulded article of the invention has an SEA value of at least 75 Jm^2/kg on impact of a 7.62 x 39 Mild Steel Core P.S. Ball M1943.

The percentage of the theoretical maximum density is hereafter referred to as the relative density. Preferably, the relative density of the compressed stack is at least 98.5%, more preferably at least 99.0% and most preferably at least 99.5%. In this way, a ballistic-resistant moulded article having an even better SEA value is obtained. More preferably, the SEA value is at least 90, even more preferably at least 110 Jm²/kg, most preferably at least 120 Jm²/kg.

The density of the ballistic-resistant moulded article can be determined in a manner known to one skilled in the art. A suitable, accurate method is

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described in ASTM D 792-91. Another suitable method for flat or slightly flexed moulded articles comprises measurement of the density by measuring the weight and accurately measuring the volume with the aid of a Vernier calliper. By this measurement, if accurately performed, the density can be determined with a standard deviation of 0.2-0.4%.

The SEA is defined by $0.5 \cdot m \cdot v_{50}^2/AD$, where m is the mass of the projectile. The v_{50} is the velocity of the projectiles fired at the moulded article at which 50% of the projectiles fully penetrate the moulded article, AD is the areal density of the moulded article.

Owing to the surprisingly high SEA achieved, not only moulded articles with an unheard-of level of protection for a given weight of the moulded article are now available but also moulded articles affording the same level of protection as the known moulded article at a significantly lower weight. Low weight per unit area is of great importance in many applications. This is the case, for instance, in the field of personal protective equipment such as helmets, shields, shoes and the like. Low weight is also essential for the application of ballistic-resistant moulded articles in for instance helicopters, motorcars and high-speed, highly manoeuvrable combat vehicles.

The areal density of the moulded article depends on the desired level of protection. Preferably, the areal density is between 10 and 40 kg/m 2 . Below 10 kg/m 2 , the level of protection is inadequate for most threats, above 40 kg/m 2 the weight will often be too high, especially for body protection.

In the context of the present application "monolayer" means a layer of substantially parallel reinforcing fibres embedded in a plastic matrix material. The term "matrix material" means a material which binds the fibres together and which wholly or

partially encapsulates the fibres. Such monolayers (usually called prepregs by one skilled in the art) and methods of obtaining such a monolayer are disclosed in for instance EP-B-0.191.306 and WO 95/00318. A monolayer may be obtained by for instance pulling a 5 number of fibres from a fibre bobbin frame over a comb so that they are oriented in coplanar and parallel fashion in one plane and then impregnating the fibres with the plastic. In this process, fibres may be used that have previously been coated with a polymer other 10 than the plastic matrix material in order to, for instance, protect the fibres during handling or in order to obtain better adhesion of the fibres onto the plastic of the monolayer. Preferably, uncoated fibres 15 are used. Impregnation of the reinforcing fibres with the plastic matrix material can be effected by applying one or more films of the plastic to the top, bottom or both sides of the plane of the fibres and then passing these, together with the fibres, through a heated pressure roll. Preferably, however, the fibres, after 20 being oriented in coplanar, parallel fashion in one plane, are coated with an amount of a liquid substance containing the plastic matrix material of the monolayer. The advantage of this is that more rapid and better impregnation of the fibres is achieved. The 25 liquid substance may be for example a solution, a dispersion or a melt of the plastic. If a solution or a dispersion of the plastic is used in the manufacture of the monolayer, the process also comprises evaporating 30

"Reinforcing fibre" here means an elongate body whose length dimension is greater than the transverse dimensions of width and thickness. The term "reinforcing fibre" includes a monofilament, a multifilament yarn, a tape, a strip, a thread, a staple fibre yarn and other elongate objects having a regular or irregular cross-section. Any natural or synthetic

the solvent or dispersant.

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fibre may in principle be used as reinforcing fibre. Use may be made of for instance metal fibres, semimetal fibres, inorganic fibres, organic fibres or mixtures thereof. For application of the fibres in ballistic-resistant moulded parts it is essential that the fibres be ballistically effective, which, more specifically, requires that they have a high tensile strength, a high tensile modulus and/or high energy absorption. It is preferred for the fibres to have a tensile strength of at least 1.2 GPa and a tensile modulus of at least 40 GPa.

Suitable inorganic fibres having a high tensile strength are for example glass fibres, carbon fibres and ceramic fibres. Suitable organic fibres having a high tensile strength are for example aramid fibres, liquid crystalline polymer fibres and fibres of for instance polyolefins, polyvinylalcohol, polyacrylonitrile which are highly oriented such as those obtained by means of a gel spinning process.

An extensive enumeration and description of fibres that may be used in the monolayer are given in WO 91/12136 (line 23 on page 6 to line 8 on page 12).

Homopolymers and copolymers of polyethylene and polypropylene are particularly suitable as polyolefins. Furthermore, the polyolefins used may contain small amounts of one or more other polymers, in particular other alkene-1-polymers.

It is preferred for the reinforcing fibres in the monolayer to be high-drawn fibres of high-molecular weight linear polyethylene. High molecular weight here means a molecular weight of at least 400,000 g/mol.

Linear polyethylene here means polyethylene having fewer than 1 side chain per 100 C atoms, preferably fewer than 1 side chain per 300 C atoms. The polyethylene may also contain up to 5 mol % of one or more other alkenes which are copolymerisable therewith, such as propylene, butene, pentene, 4-methylpentene,

octene.

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Preferably, use is made of polyethylene fibres consisting of polyethylene filaments prepared by a gel spinning process as described in for example GB-A-2042414 and GB-A-2051667. This process essentially comprises the preparation of a solution of a polyolefin of high intrinsic viscosity, spinning the solution to filaments at a temperature above the dissolving temperature, cooling down the filaments below the gelling temperature so that gelling occurs and drawing the filaments before, during or after removal of the solvent.

The shape of the cross-section of the filaments can be selected here through selection of the shape of the spinning aperture.

Preferably, the monolayer contains reinforcing fibres, preferably the aforementioned strong polyethylene fibres, with a denier per filament (dpf) greater than or equal to 1.5. Most preferably, use is made of multifilament yarns of ultrahigh molecular weight linear polyethylene with an intrinsic viscosity of at least 5 dl/g, determined in decalin at 135°C, and a yarn titre of at least 50 denier, with the yarn having a tensile strength of at least 35 cN/dtex and a tensile modulus of at least 1000 cN/dtex and the filaments having a cross-section aspect ratio of at most 3. The use of these fibres has been found to improve the high level of protection of the ballistic-resistant panel of the invention still further.

The plastic matrix material may wholly or partially consist of a polymer material, which optionally may contain fillers usually employed for polymers. The polymer may be a thermoset or thermoplastic or mixtures of both. Preferably a soft plastic is used, in particular it is preferred for the plastic matrix material to be an elastomer with a tensile modulus (at 25°C) of at most 41 MPa.

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Preferably, the elongation to break of the plastic is greater than the elongation to break of the reinforcing fibres. The elongation to break of the matrix preferably is from 3 to 500%.

Thermosets and thermoplastics that are suitable for the monolayer are listed in for instance WO-A-91/12136 (line 26, page 15 to line 23, page 21). Preferably, vinylesters, unsaturated polyesters, epoxides or phenol resins are chosen as matrix material from the group of thermosetting polymers. These thermosets usually are in the monolayer in partially set condition (the so-called B stage) before the stack of monolayers is cured during compression of the ballistic-resistant moulded article. From the group of thermoplastic polymers polyurethanes, polyvinyls, polyacryls, polyolefins or thermoplastic, elastomeric block copolymers such as polyisoprene-polyethylenebutylene-polystyrene or polystyrene-polyisoprenepolystyrene block copolymers are preferably chosen as matrix material.

The plastic-matrix-material content of the monolayer is chosen sufficiently low, in any case lower than 30 wt.% relative to the total weight of the monolayer. Preferably, the content is lower than 20 wt.%, most preferably lower than 10 wt.%.

The fibre direction in each monolayer in the ballistic-resistant moulded article of the invention is rotated with respect to the fibre direction in an adjacent monolayer. This construction is hereafter referred to as being "cross-layered". Good results are achieved when this rotation amounts to at least 45 degrees. Preferably, this rotation amounts to approximately 90 degrees.

The relatively high density and an SEA value of at least $75 \, \mathrm{Jm^2/kg}$ of the compressed stack of the invention can be achieved by taking a number of measures. It is possible for instance to ensure in a

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number of stages in the manufacture of the moulded articles that as few low-density components, like air or from the polymer evaporated gasses as possible remain in the moulded article. This can for instance be accomplished by using a plastic of very low viscosity in impregnating the reinforcing fibres in the manufacture of the monolayer. It is also advantageous to spread the reinforcing fibres well during the impregnation process or to subject them to for instance ultrasonic vibration. As mentioned above, if multifilament yarns are used, it is important for a good spread that the yarns have a low twist. Furthermore, the plastic may be applied in vacuo. Each of these measures has a beneficial effect on the level of protection of a ballistic-resistant moulded article obtained with the monolayers but the level of protection of the moulded article of the invention is not attained or is attained only with great effort.

The invention also relates to a process for the manufacture of a ballistic-resistant moulded article in which monolayers are stacked cross-wise, whereupon the stack is compressed at a given pressure at an elevated temperature.

A process of this kind is disclosed in for instance WO 89/06190. It teaches that when the plastic matrix material is heated to its melting point, relatively low pressures are sufficient for shaping the moulded article. The pressures stated vary from 0.05 MPa to a maximum of 3 MPa.

It has now surprisingly been found that significantly higher SEA values than hitherto known in the state of the art can be attained by compressing and cooling the stack at a pressure of at least 5 MPa.

The pressure preferably is at least 7 MPa. In this way, the theoretical maximum density of the compressed stack is approached more closely and the level of protection against the impact of projectiles

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is increased further. The pressure preferably is at least 10 MPa, more preferably at least 13 MPa and most preferably at least 15 MPa. Preferably, the pressures during compression at an elevated temperature and during cooling are the same.

Compression at an elevated temperature is intended to mean that the moulded article is subjected to the given pressure for a particular compression time at a compression temperature above the softening or melting point of the plastic matrix material and below the softening or melting point of the fibres. The required compression time and compression temperature depend on the kind of fibre and matrix material and on the thickness of the moulded article and can be readily determined by one skilled in the art.

It has been found that in order for a high SEA value to be attained it is necessary that after compression at an elevated temperature cooling, too, take place under pressure.

Cooling under pressure is intended to mean that the given minimum pressure is maintained during cooling at least until so low a temperature is reached that the structure of the moulded article can no longer relax under atmospheric pressure so that the SEA value cannot decrease. This temperature is easy to establish by one skilled in the art. It is preferred for cooling at the given minimum pressure to be down to a temperature at which the plastic matrix material has largely or completely hardened or crystallized and below the relaxation temperature of the reinforcing fibres. The pressure during the cooling does not need to be equal to the pressure at the high temperature, but it is preferred that these pressures are equal. The pressure loss resulting from shrinkage of the moulded article and the press due to cooling must regularly be compensated for so as to keep the pressure during

cooling constant or at least on a sufficient high

AMENDED PAGE 10

level.

As the stack of monolayers is heated to the desired compression temperature, the pressure preferably is increased to the desired level in, preferably at least three, discontinuous steps. In this way, blisters are prevented from forming.

For the manufacture of a ballistic-resistant moulded article in which the reinforcing fibres in the monolayer are high-drawn fibres of high-molecular-weight linear polyethylene, the compression temperature is preferably 115 to 130°C and cooling to below 70°C is effected at a constant pressure. Compression temperature here means the temperature at half the thickness of the moulded article.

In the process of the invention the stack may be made starting from loose monolayers. Loose monolayers are difficult to handle, however, in that they easily tear in the fibre direction. It is therefore preferred to make the stack from consolidated monolayer packages containing from 2 to 8, as a rule 2, 4 or 8, monolayers that are placed cross-wise. Consolidated is intended to mean that the monolayers are firmly attached to one another. Very good results are achieved if the monolayer packages, too, are compressed at an elevated temperature and cooled at a high pressure, preferably of at least 5 MPa.

US-3743561 describes a process for the manufacturing of composite structures or articles from reinforcing fibers in a two step process. In the first step an intermediate structure is manufactured. In a second step a number of intermediate articles are assembled to form the composite article. Although US-3743561 describes the objective of improving mechanical properties, no disclosure is given about ballistic properties of the structures or articles.

In another embodiment of the process of the invention the ballistic-resistant moulded article is manufactured by a process whereby, in a first step, a semi-manufactured

AMENDED PAGE 10A

article is produced by forming a stack of cross-layered monolayers, which stack has an areal density of from 0.25 to 5 $\rm kg/m^2$ and subsequently compressing this stack at an elevated temperature and cooling this stack at a given first pressure whereupon, in a second step, the semi-manufactured articles are compressed onto each other at an elevated temperature and cooled at a given second pressure, with the first

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pressure being at least 5 MPa.

An advantage of this process is that, if the semi-manufactured articles are also compressed and cooled at a pressure of at least 5 MPa in the second step, the moulded articles obtained have a higher SEA value than moulded articles manufactured in a single compression step at the same pressure.

Another important advantage of the abovementioned process is that high SEA values are
attainable when the second pressure is relatively low,
preferably at most 5 MPa. It has been found that good
SEA values can be attained if the second pressure is
lower than 5 MPa or even 3 MPa. A major advantage of
this is that manufacturers of ballistic-resistant
moulded articles, who often do not have high pressure
moulds (i.e. higher than 5 MPa) at their disposal, can
still achieve the good properties of the ballisticresistant moulded article of the invention using
conventional moulding equipment.

The semi-manufactured articles can then be centrally produced in locations where a high-pressure mould is available, for example at the manufacturer of the monolayers or monolayer packages.

The invention also relates to the semi25 manufactured article for the manufacture of a
ballistic-resistant moulded article of the invention
that is obtainable by the first step in the abovementioned process.

The semi-manufactured article has an areal

density of from 0.25 to 5 kg/m². An areal density lower
than 0.25 kg/m² has the disadvantage that the SEA value
of the ballistic-resistant moulded article ultimately
obtained is lower. An areal density higher than 5 kg/m²
has the disadvantage that there is little flexibility

in the choice of the areal density of the ballisticresistant moulded article ultimately to be formed and
that the cycle time for compressing the semi-

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manufactured article becomes unacceptably long. Preferably, the areal density of the semi-manufactured article is between 0.5 and 2.5 kg/m^2 .

It has surprisingly been found that, using the process of the invention, higher SEA values are attainable if the reinforcing fibres in the monolayers have a cross-section aspect ratio of at most 3. Cross-section aspect ratio is intended to mean the ratio of the longest to the shortest principal axis of a perpendicular cross-section of the fibre. Preferably, the aspect ratio is lower than 2, more preferably lower than 1.5. It has been found that high SEA values are obtained at low compressive pressures in ballistic-resistant moulded articles in which the filaments have a lower aspect ratio (being rounder). For the same reason, the filaments preferably also have a fineness of 1.5 denier per filament or more.

Furthermore, it has been found that, using the process of the invention, higher SEA values are attainable at comparable compressive pressures if the monolayers employed in the ballistic-resistant moulded article are obtained by impregnating reinforcing fibres with an aqueous dispersion which contains the plastic matrix material.

Furthermore, it has been found that, using the process of the invention, higher SEA values are attainable at comparable compressive pressures if the monolayers employed in the ballistic-resistant moulded article have a fibre weight higher than 25, more preferably higher than 50 g/m². Preferably, the fibre weight is at most 150 g/m², more preferably lower than 100 g/m^2 .

The invention is illustrated by means of the following examples without being limited thereto.

Examples I and II and comparative experiments A and B

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Material used:

A monolayer package consisting of four monolayers disposed cross-wise at an angle of 90 degrees. The fibres are high-drawn fibres of high molecular weight linear polyethylene of make Dyneema SK66 (TM) with a strength of 32.5 cN/dtex, a modulus of 1015 cN/dtex and a fineness of 1 denier per filament with a cross-section aspect ratio of about 5. The monolayer contains 20 wt.% matrix material consisting of Kraton (TM) applied from an aqueous dispersion. Kraton is a styrene-isoprene-styrene triblock copolymer. The areal density of the monolayer package is 135 g/m².

15 Procedure:

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A number of the above-mentioned monolayer packages were stacked to yield a package whereupon the package in its entirety was placed between two platens of a standard press. The temperature of the platens was about 125-130°C. The package was retained in the press until the temperature at the centre of the package was 115-125°C. Subsequently, the pressure was increased to a compressive pressure as stated in Table 1 below and the package was kept under this pressure for 15 min. Next, the package was cooled to a temperature of 60°C at the same compressive pressure.

Test procedure:

The panels obtained were fired at in

30 accordance with the procedure set out in STANAG 2920.

Use was made of a 7.62 x 39 mm Mild Steel Core (PS Ball M 1943) bullet weighing 8 grammes. The bullet used had been obtained from Messrs Sellier & Bellot, Czech Republic. The experimental density of the panels was determined by accurate weighing and measurement of the volume using a Vernier calliper. The standard deviation according to this density measurement is 0.002-0.004

 g/cm^3 .

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Results:

The v_{50} and SEA values as a function of the compressive pressure are given in Table 1. The stated SEA value was calculated from the total areal density of the ballistic-resistant moulded article. Relative density is intended to mean the ratio of the experimental density of the panel ρ_p and the theoretical density of the panel, which is defined by $(\rho_f \ m_f + \rho_k m_k)$. The theoretical density of the ballistic-resistant moulded article as calculated from $\rho_f = 0.97$, $m_f = 0.8$ and $\rho_k = 0.943$ amounts to 0.965.

15 Table 1

	AD (kg/m²)	compressiv e pressure (MPa)	V ₅₀ (m/sec)	SEA (Jm²/kg)	Relative density
A	33.5	1.0	591	42	0.969
В	33.5	2.5	668	53	0.974
I	33.8	7.0	825	81	0.990
II	26.9	16.5	772	89	0.992

The results indicate that, at equal areal density, the ballistic-resistant panel of the invention has a surprisingly higher SEA value than a panel with a lower rel. density. The table also shows that the panel of the invention can be obtained by choosing a compressive pressure of at least 5 MPa.

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Examples III-VI

Material used:

A monolayer package consisting of four 5 monolayers disposed cross-wise at an angle of 90 degrees. The monolayer package differs from the monolayer packages referred to in Examples I and II only in respect of the fibres used. The fibres are high-drawn fibres of high molecular weight linear polyethylene with a yarn titre of 880 denier, a tensile 10 strength of 37.3 cN/dtex, a tensile modulus of 1220 cN/dtex, an elongation to break of 3.7%, a fineness of 2 denier per filament and a round cross-section aspect ratio of about 1. The monolayer contains 20 wt.% matrix material consisting of Kraton (TM). The areal density 15 of the monolayer package is 135 g/m^2 .

Procedure and testing

Starting from the above-mentioned monolayer packages, ballistic-resistant moulded articles were produced at various compressive pressures and tested in the manner set out in Example 1. The ballistic-resistant panel obtained had an areal density equal to 25.9 kg/m² for all compressive pressures.

The results are given in Table 2.

Table 2

Example	compressive pressure (MPa)	V ₅₀ (m/sec)	SEA Jm²/kg)	relative density (-)
III	1.0	741	85	0.981
IV	2.5	768	91	0.988
٧	7.0	913	129	0.996

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Example	compressive pressure (MPa)	V ₅₀ (m/sec)	SEA Jm²/kg)	relative density (-)
VI	16.5	939	136	0.995

Comparative Experiment C and Examples VII and VIII

In Comparative Experiment C and Examples VII and VIII, ballistic-resistant moulded articles were produced from monolayer packages as described in Example I in the manner described above. The compressive pressures in Comparative Experiment C and Example VII were 1 MPa and 7 MPa, respectively. In Example VIII, semi-manufactured articles consisting of 5 monolayer packages were first moulded at a first compressive pressure of 5 MPa, which semi-manufactured articles were then stacked and compressed at a second pressure of 1 MPa. In each case, the compressive pressure was kept at the same level during cooling. The results are given in Table 3.

Table 3

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	AD (kg/m²)	compressive pressure (MPA)	V ₅₀ (m/sec)	SEA (Jm²/kg)
С	29.6	1	614	51
VII	33.8	7	825	81
VIII	29.1	5/1	739	75

Comparative Experiments D and E and Examples IX and X

As the first starting material (S1) a monolayer package available under the name of SpectraShield (TM) and consisting of four monolayers

disposed crosswise at an angle of 90 degrees. The fibres are high-drawn fibres of high-molecular-weight linear polyethylene of make Spectra 1000 (TM) with a fineness of 5.5 denier per filament and a cross-section aspect ratio of about 1. The monolayer was obtained by impregnating the unidirectional fibres with a solution of the matrix material Kraton (TM).

As the second starting material (S2) a

10 monolayer package was prepared by first preparing a
monolayer starting from Spectra 1000 fibres which were
impregnated with an aqueous dispersion (instead of a
solution as in S1) of the matrix material Kraton. The
yarn weight in the monolayer is 52 g/m², the matrix

15 content is 20%. Of these monolayers four-layer
monolayer packages were made.

The monolayer packages S1 and S2 were stacked and compressed into ballistic-resistant moulded articles as described above. In Comparative Experiment D and Example IX starting material S1 was used, in Comparative Experiment E and Example X starting material S2 was used. The compressive pressures used and the results are given in Table 4.

Table 4

Examp	ole	AD (kg/m²)	compressive pressure (MPA)	V ₅₀ (m/sec)	SEA (Jm²/kg)
D	(S1)	24.9	1	524	44
E	(S2)	22	1	598	65
IX	(S1)	24.9	5	761	93
х	(S2)	22	5	792	114

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A comparison between Comparative Experiments D and E and Examples IX and X shows the great improvement in ballistic resistance. A comparison of the starting materials S1 and S2 in Examples VII and VIII shows the great improvement in ballistic resistance obtained when the monolayers have been prepared with an aqueous dispersion of the matrix material in combination with high-pressure compression.

AMENDED CLAIMS

- Ballistic-resistant moulded article containing a compressed stack of monolayers, with each monolayer containing unidirectionally oriented reinforcing fibres and at most 30 wt.% of a plastic matrix material and with the fibre direction in each monolayer being rotated with respect to the fibre direction in an adjacent monolayer, characterized in that the density (ρ_P) of the compressed stack is at least 98.0% of the theoretical maximum density.
- 2. Ballistic-resistant moulded article according to claim 1, characterized in that the density ρ_P is at least 99.0% of the theoretical maximum density.
- 3. Ballistic-resistant moulded article according to claim 1 or claim 2, characterized in that the reinforcing fibres in the monolayer have a tensile strength of at least 1.2 GPa and a tensile modulus of at least 40 GPa.
- 4. Ballistic-resistant moulded article according to any one of the claims 1-3, characterized in that the reinforcing fibres in the monolayer are high-drawn fibres of high-molecular-weight linear polyethylene.
- 5. Ballistic-resistant moulded article according to claim 1, characterized in that the plastic matrix material is an elastomer with a tensile modulus (at 25°C) of at most 41 MPa.
- 6. Ballistic-resistant moulded article according to claim 1, characterized in that the rotation amounts to 90 degrees.
- 7. Ballistic-resistant moulded article according to claims 1-6, characterized in that the moulded article has a specific energy absorption (SEA) of at least 75 Jm^2/kg on impact of a 7.62 x 39 Mild Steel Core P.S. Ball M1943 bullet.
- 8. Process for manufacturing a ballistic resistant moulded article in which a stack is made of crosswise-arranged monolayers, which stack is compressed under pressure of at least 5 MPa and at an elevated temperature and which

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AMENDED CLAIMS

stack is thereafter cooled under pressure, characterized in that the monolayers have a fiber weight between 25 and 100 gr/m^2 .

- 9. Process for manufacturing a ballistic-resistant moulded article in which in a first step a semi-manufactured article is produced by forming a stack of cross-layered monolayers, which stack has an areal density of from 0.25 to 5 kg/m², and subsequently compressing this stack at an elevated temperature and cooling this stack at a given first compressive pressure whereupon, in a second step, the semi-manufactured articles are compressed onto each other at an elevated temperature and cooled at a given second pressure, with the first pressure being at least 5 MPa.
- 10. Process according to claim 9, characterized in that the second pressure is at most 5 MPa.
- 11. Process according to claim 8 or 9, characterized in that the stack is made from consolidated monolayer packages containing from 2 to 8 monolayers that are placed crosswise.
- 12. Process according to claim 11, characterized in that the monolayer packages, too, are compressed and cooled at a pressure of at least 5 MPa.
- 13. Process according to any one of the claims 8, 9 or 12, characterized in that the specified pressure is at least 7 MPa.
- 14. Process according to any one of the claims 8, 9 or 12, characterized in that the specified pressure is at least 10 MPa.
- 15. Process according to any one of the claims 8-14, characterized in that the reinforcing fibres in the monolayers have a cross-section aspect ratio of at most 3.
- 16. Process according to any one of the claims 8-15, characterized in that the monolayer has been obtained by im

AMENDED CLAIMS

pregnating the reinforcing fibres with an aqueous dispersion containing the plastic matrix material.

- 17. Process according to any one of the claims 8-16, characterized in that the monolayer has a fibre weight of between 50 and 150 g/m^2 .
- 18. Ballistic-resistant moulded article obtainable according to the process of any of the cliams 8-17, characterized in that the moulded article has a SEA of at least 75 Jm²/kg on impact of a 7.62 Mild Steel Core P.S. Ball M1943 bullet.
- 19. Semi-manufactured article according to the first step in the process according to claim 9 for the manufacture of a ballistic-resistant moulded article according to any of the claims 9-16, characterized in that the areal density of the semi-manufactured article is between 0.25 and 5 kg/ m^2 .

ABSTRACT

The invention relates to a ballistic-resistant moulded article containing a compressed stack of monolayers, with each monolayer containing unidirectionally oriented reinforcing fibres and at most 30 wt.% of a plastic matrix material and with the fibre direction in each monolayer being rotated with respect to the fibre direction in an adjacent monolayer, characterized in that the density (ρ_P) of the compressed stack is at least 98.0% of the theoretical maximum density. The invention also relates to a process for manufacturing the moulded article.

The ballistic-resistant article may be used in, for instance, helmets, as inserts in bullet-proof vests, as armouring on military vehicles and in ballistic-resistant panels.

FOR UTILITY/DESIGN CIP/PCT NATIONAL/PLANT ORIGINAL/SUBSTITUTE/SUPPLEMENTAL DECLARATIONS

RULE 63 (37 C.F.R. 1.63) DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

CUSHMAN FORM

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name, and I

believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed low) of the subject matter which is claimed and for which a patent is sought on the INVENTION ENTITLED LALLISTIC-RESISTANT MOULDED ARTICLE AND A PROCESS FOR THE MANUFACTURE OF THE MOULDED ARTICL the specification of which (CHECK applicable BOX(ES)) is attached hereto. as U.S. Application No. mas filed on Х 100247 on 14 June M was filed as PCT International Application No. PCT/ NL96 BOX(ES) 29 July 1997 → and (if applicable to U.S. or PCT application) was amended on I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56. I hereby claim foreign priority benefits under 35 U.S.C. 119/365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate filed by me or my assignee disclosing the subject matter claimed in this application and having a filing date (1) before that of the application on which priority is claimed, or (2) if no priority claimed, before the filing date of this application: **Date Patented Priority Claimed** PRIOR FOREIGN APPLICATION(S) Date first Laid-Day/MONTH/Year Filed No open or Published or Granted <u>Yes</u> Number Country Q 23 December 1996 20 June 1995 1000598 the Netherlands I hereby claim domestic priority benefit under 35 U.S.C. 119/120/365 of the indicated United States applications listed below and PCT international applications listed above or below and, if this is a continuation-in-part (CIP) application, insofar as the subject matter disclosed and claimed in this application is in addition to that disclosed in such prior applications, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56 which became available between the filing date of each such prior application and the national or PCT international filing date of this application: PRIOR U.S. PROVISIONAL, NONPROVISIONAL AND/OR PCT APPLICATION(S) **Priority Claimed** <u>Status</u> m Yes <u>No</u> Application No. (series code/serial no.) Day/MONTH/Year Filed pending, abandoned, patented 14 June 1996 pending --- PCT/NL96/00247 13 I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon. And I hereby appoint Cushman Darby & Cushman Intellectual Property Group of Pillsbury Madison & Sutro LLP, 1100 New York Avenue, N.W., Ninth Floor, East Tower, Œ Washington, D.C. 20005-3918, telephone number (202) 861-3000 (to whom all communications are to be directed), and the below-named persons (of the same address) u individually and collectively my attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith and with the resulting patent, and I hereby authorize them to delete names/numbers below of persons no longer with their firm and to act and rely on instructions from and communicate directly with the person/assignee/attomey/firm/ organization who/which first sends/sent this case to them and by whom/which I hereby declare that I have consented after full disclosure to be represented unless/until I instruct the above Firm and/or a below attorney in writing to the contrary. 31097 Mark G. Paulson 30793 David W. 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Prince 22429 Date: 10-12di (1) INVENTOR'S SIGNATURE: con L.H. der Loo van Leonardus **Family Name** Middle Initial First the Netherlands the Netherlands Residence Beek Country of Citizenship State/Foreign Country City Beek, the Netherlands 6176 DK Post Office Address Iepenlaan (include Zip Code) Date: (2) INVENTOR'S SIGNATURE: Family Name Middle Initial First Residence Country of Citizenship State/Foreign Country City Post Office Address (include Zip Code)

(FOR ADDITIONAL INVENTORS, check box ☐ to attach CDC 116-2 same information for each re signature, name, date, citizenship, residence and address.)